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RECENTLY PUBLISHED RESEARCH OF THE
LENINGRAD INSTITUTE OF CHEMICAL TECHNOLOGY, USSR

"Reaction of Phthalic Anhydride With Acenaphthene in the Presence of Aluminum Chloride," Z. A. Veynberg, Leningrad Chem Tech Inst

"Zhur Obshch Khim" Vol 17, 1947, pp 1662-70

The product of the reaction of acenaphthene with $C_6H_4(CO)_2O$ and $AlCl_3$ described in US Patent 1,997,305 was investigated. The reaction is shown to go through the initial formation of acenaphthoylbenzoic acid, which then loses water and forms the final 4,5-phthaloylacenaphthene.

"Transformations of Pinacole With Substituted Acetylene Radicals: III, Synthesis and Transformations of Dimethylphenyl (Tert-Butylethynyl)-Ethylene Glycol," E. D. Venus-Danilova, E. P. Brichko, Leningrad Chem Tech Inst

"Zhur Obshch Khim" Vol 17, 1947, pp 1549-53

To the Grignard reagent, prepared conventionally, was added $MgC(OH)Br$ in Et_2O ; after decomposition with dilute acid there was obtained 75.5% dimethylphenyl (tert-butylethynyl)ethylene glycol (I), $MgC(OH)CPh_2$ ($OR/C:CCMe_3$). I and 20% H_2SO_4 boiled with stirring, and extracted with Et_2O gave 2,6,6-trimethyl-3-phenyl-3-hepten-2-ol-5-one (II), while extraction with Et_2O of the neutralized aqueous layer from the first

- 1 -

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extraction gave 2-tert-butyl-4-phenyl-5,5-dimethyl-2-hydroxy-2,5-dihydrofuran (III); a similar reaction using 30% H_2SO_4 gave slightly lower II and a greater amount of III. Both II and III react with MgI_2 ; II decolorizes $KMnO_4$ solution and Br solution in $CHCl_3$, while III does not do so. II gives initially a red color with concentrated H_2SO_4 ; III yields a yellow precipitate, soluble in excess H_2SO_4 with blue-green fluorescence. II and III form identical semicarbazones, but the reaction with III must be catalyzed with a trace of $AcOH$. II with 2% $KMnO_4$ gave Me_2CO , $EtOH$ and Me_2CCO_2H . III, $EtOH$, and $AcOH$ heated to boiling and allowed to stand gave the 2-EtO derivative, which hydrolyzes readily on standing in air to the original III. A similar reaction using II gave the starting material and small amounts of tar.

"Hydro- and Solvolytic and Intramolecular Oxidation-Reduction: I, Acetolysis of Cellulose as Solvolytic Oxidation-Reduction," S. N. Danilov, P. T. Pastukhov, Leningrad Chem Tech Inst

"Zhur Obshch Khim" Vol 17, 1947, pp 1140-61

Acetolysis of bleached cotton linter and of triacetylcellulose was accomplished by the procedure of Fieser and Hess at 300° , in the presence of H_2SO_4 , 75% and 30-75% of the weight of the initial I and II, respectively. Determinations of viscosity and of the iodine number and % $AcOH$ in the precipitated product were made. Along with the obvious determinations of melting point, solubility, rotatory power, $AcOH$ and iodine numbers, the composition of the product at various stages was investigated by the bromoacetate derivatives following the method of Fieser; $HBr + AcOH$ acts only on monosaccharides and on oligosaccharides, not on cellulose or on cello-dextrins. Separation of the various fractions after treatment with $HBr + AcOH$, in particular, isolation of the cellobiose octaacetate derivative, is facilitated by the use of $BClO_3$ instead of H_2SO_4 , owing to the absence of mixed inorganic-organic esters with $BClO_3$. On the basis of experimental facts, and of the necessity of the presence of H_2O for acetolysis, the most likely reaction scheme is $(C_6H_{10}O_5)_n + nAc_2O \rightarrow (n/2) (C_{12}H_{14}O_7)(OAc)_2 + 4nAcOH$. The role of H_2SO_4 consists in formation of acetyl-sulfuric acid which forms a glucoside link with the bridge C of cellulose; the H_2SO_4 is subsequently split off by H_2O ; this hydrolytic action is evidently catalyzed by acid.

"Isomerization of Hydroxy Aldehydes: VII, Oxidative-Reductive Transformation of Alpha-Hydroxybutyraldehyde," E. D. Venus-Danilova, V. F. Kazimirova, Leningrad Chem Tech Inst

"Zhur Obshch Khim" Vol 16, 1946, pp 2099-2103

In acid solution $EtCH(OH)CHO$ (I) is transformed into $MeCH(OH)Ac$ and its oxidation product, Ac_2 , which gave in part

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an aldolization product, 3-methylheptan-3-ol-2,5,6-trione (II). Some $\text{EtCH(OH)CO}_2\text{H}$ (III) was also formed. Both in acid media and in the presence of weak alkali with Cu(OH)_2 or Pb(OH)_2 as catalysts, the sole primary product of I is MeCH(OH)Ac . These transformations are unexplainable by the oxide-intermediate mechanism, which would predict the formation of a primary rather than the actually observed secondary alcohol. It is possible to explain the reaction by an enolization mechanism: $\text{RCH}_2\text{CH(OH)CHO} \rightarrow \text{RCH}_2\text{C(OH):CHOH} \rightarrow \text{RCH:C(OH)CH}_2\text{OH} \rightarrow \text{RCH(OH)C(OH):CH}_2 \rightarrow \text{RCH(OH)COCH}_3$.

"Reaction Between Aromatic Diamines and Dicarboxylic Acids: VI, Reaction of Tolidine and Dianisidine With Phthalic Anhydride," B. A. Poray-Koshits, D. A. Bodik, Dyestuff Lab, Leningrad Chem Tech Inst, Moscow

"Zhur Obshch Khim" Vol 15, 1945, pp 245-51

It is shown that ortho-tolidine or ortho-dianisidine condense with one molecule phthalic anhydride (I) to yield monophthaloyl derivatives without formation of "H" bonds between N and O atoms. Thus, the Kauffler formula with biplanar structure of these compounds is disproved. Neither monophthaloyl derivative is capable of dehydration to form a substance analogous to Gulis-melli's "imidazole." Both derivatives form diphtaloyl derivatives on further reaction with I.

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- 3 -

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